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January 27, 2014

Ms. Candice Teichert  
EPA Region 4 Superfund Division  
61 SW Forsyth Street, 11th Floor, Atlanta Federal Center  
Atlanta, Georgia 30303

Subject: Sampling and Analysis Plan - Addendum 2, Rev 0  
Barite Hill/Nevada Goldfields Site  
McCormick County, South Carolina

Dear Ms. Teichert:

Enclosed are three (3) copies of the Sampling and Analysis Plan - Addendum 2, Rev 0 for the Barite Hill Goldfields site located in McCormick, South Carolina.

If you have any questions, or require any additional information, please call me at (770) 521-8119.

Sincerely,

**BLACK & VEATCH Special Projects Corp.**

John Jenkins  
Task Order Manager

cc: Michael Allen, USEPA (w/o enclosure)  
Charles Swan, USEPA (w/o enclosure)  
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**Sampling and Analysis Plan - Addendum 2  
Revision 0**

**Barite Hill/Nevada Goldfields Site  
Remedial Investigation and Feasibility Study  
McCormick County, South Carolina  
CERCLIS ID SCN000407714**

**Prepared Under:  
EPA Contract EP-S4-09-02  
EPA Task Order No. 038-RICO-A4NZ  
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DCN: 49038-0101-02-D-01649R0A2**

**Prepared for:  
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**January 2014**

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## Acronyms and Abbreviations

Black & Veatch	Black & Veatch Special Projects Corp.
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
DO	dissolved oxygen
EPA	U.S. Environmental Protection Agency
FS	Feasibility Study
FSP	Field Sampling Plan
mg/L	milligrams per liter
NPL	National Priority List
pH	hydrogen ion concentration
ORP	oxidation-reduction (redox) potential
OTIE	Oneida Total Integrated Enterprises
PVC	polyvinyl chloride
QAPP	Quality Assurance Project Plan
RI	Remedial Investigation
SAP	Sampling and Analysis Plan
SESD	Science and Ecosystems Support Division
Site	Barite Hill /Nevada Goldfields Site
SOP	Standard Operating Procedure
START	Superfund Technical Assessment and Response Team
TO	Task Order
TOC	total organic carbon
µm	micrometre



## 1.0 Introduction

This document comprises the *Sampling and Analysis Plan (SAP) Addendum 2, Revision 0* to the *Barite Hill Goldfields Site, Sampling and Analysis Plan (SAP), Volumes 1 and 2, Revision 1*, prepared by Black & Veatch Special Projects Corp. (Black & Veatch), and dated April, 2011. This *Addendum 2, Revision 0* details activities to be performed by Black & Veatch in support of the Remedial Investigation (RI)/Feasibility Study (FS) for the Barite Hill /Nevada Goldfields Site (Site).

The Site is a former gold and silver mining facility located in McCormick County, South Carolina approximately three miles southwest of the town of McCormick. The property covers approximately 795 acres, of which about 135 acres has been disturbed by historic and modern mining. The Site, which was most recently operated from about 1991 to 1995, was partially reclaimed prior to being abandoned by the operator in 1999. The Barite Hill mine was operated historically through underground workings and more recently as an open-pit and cyanide heap leach facility. When the mine was abandoned, pumping of water in the Main Pit ceased and the pit began to flood, eventually forming an acidic lake approximately 10 acres in size. By 2007, the lake contained approximately 100 million gallons of water with a hydrogen ion concentration (pH) of between 2.0 and 2.2 and elevated concentrations of dissolved metals. For example, the concentrations of cadmium, copper, and zinc in the Main Pit Lake were 1.57, 287, and 40.2 milligrams per liter (mg/L), respectively. The U.S. Environmental Protection Agency (EPA) initiated a time-critical removal action in 2008. This action included treating the pit water to near neutral pH, grading and covering waste rock dumps that were a major source of acidity to the Main Pit Lake and constructing a spillway to control releases to the northern unnamed tributary of Hawe Creek. Seeps from the Main Pit Lake containing acidic water with high dissolved metal content continue to discharge to the northern unnamed tributary of Hawe Creek, while waste rock dumps surrounding the eastern and southeastern portions of the Main Pit Lake continue to be a source of acid rock drainage. A full description of the operational and regulatory history of the Site was provided in the April 2011 *SAP* (Black & Veatch, 2011).

The Site was placed on the EPA National Priorities List (NPL) on April 9, 2009 (EPA, 2010a), and several environmental studies have been conducted on the Site related to actions pursuant to the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA).

The EPA Region 4, Waste Management Division, Superfund Remedial & Site Evaluation Branch began a RI/FS of the Site in 2010. A Work Plan for the site investigation was approved in August 2010 (Black & Veatch, 2010), and a Site-wide SAP that included a Field Sampling Plan (FSP) and accompanying Quality Assurance Project Plan (QAPP) was developed by Black & Veatch (Black & Veatch, 2011) for the RI/FS. The EPA Science and Ecosystems Support Division (SESD), Athens, Georgia was tasked to lead the field investigations with Black & Veatch support. A series of QAPPs were developed by SESD that paralleled the overall SAP. The SESD QAPPs included sampling of soil, mining-related wastes, surface water, sediment, and groundwater in existing monitoring and potable wells. In addition SESD prepared a QAPP for storm water sampling and conducted limited benthic surveys. Black & Veatch also had their subcontractor complete geophysical surveys in selected portions of the Site where leachate plumes may occur.

## **2.0 Scope of Additional Data Collection**

The results of Barite Hill pit lake sampling and investigations were reviewed, including vertical profiles of field parameters determined from handheld sonde drops by the EPA, the Superfund Technical Assessment and Response Team (START), SESD, and Black & Veatch and analytical results for samples collected by the START contractor Oneida Total Integrated Enterprises (OTIE) and Black & Veatch. Recommendations for future sampling and monitoring were made with respect to current understanding (Black & Veatch, 2013).

This SAP addendum describes the additional sampling that will be conducted. It is anticipated that data collection will be conducted monthly beginning in 2014, and for two rainfall events.

Because data collection will be similar to that previously gathered at the Site, modifications to the Site-wide *QAPP* (Black & Veatch, 2011) will not be required. All sampling protocols described herein will be conducted in accordance with the SESD's established *QAPPs* (EPA, 2011a-f and EPA, 2013) and Standard Operating Procedures (SOPs) and applicable Black & Veatch procedures.

Additional data needs include:

- Monthly vertical profiles of field parameters, coupled with field measurements of total and ferrous iron, total alkalinity, and total organic carbon from selected depths;
- A transducer will be obtained to measure pit lake elevation levels and data collected monthly;
- Measure vertical profiles and collect field measurements of total and ferrous iron, total alkalinity, and total organic carbon from selected depths following two rain storms;
- Collect discharge and quality of water entering the pit from each of the three storm runoff diversions, during two rain storms;
- Measure rainfall pH at the beginning of a rain event and after rain has fallen continuously for about 30 minutes; and
- Collect analytical samples of water running off the pit walls.

### 3.0 Sampling Objectives, Approach, and Requirements

The following sections describe the sampling objectives, approach, and requirements for each of the data needs identified in Section 2.0. All other aspects of the April 2011 *SAP Volumes 1 (QAPP) and 2* pertaining to field documentation, decontamination, data acquisition and management, quality control, data validation and other requirements continue to apply to this FSP Addendum.

#### 3.1 Pit Lake Water Sampling

Conduct routine monthly pit water elevations and vertical profiles (at 5 foot intervals) of field parameters, coupled with field measurements of total and ferrous iron, total alkalinity, and total organic carbon from selected depths. The purpose is to evaluate variability in pit lake chemistry that will be used to support potential remedial alternatives, as well as to record the pit water elevation daily.

**Objective:** Provide additional field and analytical data for surface water from the pit lake.

**Approach:** Routine pit lake sampling. Grab samples of surface water will be used to provide field and analytical data for these locations. Parameters to be analyzed for are provided in Table 3-2.

For one year, monthly trips will be made to the Site, and a sonde and data logger equipped with calibrated probes for pH, specific conductance, temperature, redox potential (ORP), and dissolved oxygen (DO) will be lowered vertically through the water column of the pit lake at two locations. Readings will be taken every five feet and recorded. Abrupt changes in field parameters with depth may be interpreted as chemoclines. Once the chemocline depth is roughly delineated at each location, one sample will be taken above the layer for testing (total organic carbon [TOC], iron, and alkalinity); and one sample will be taken below the layer for testing, for a total of two samples per pit lake location. See Attachment 1 for iron test kit SOP; and Attachment 2 for dilution procedures for field iron samples only (Hach Field Testing).

On one of the Site visits, a bathymetry survey will be conducted, by using a grid pattern on a boat-mounted depth sounder. This will provide data to allow a calculation of the quantity of water in the pit lake that can be tied to the measured pit lake elevation.



In addition, the water elevation will be recorded daily from one location via pressure transducer with data logger in the pit lake. Black & Veatch will be responsible for the installation, maintenance, and data collection from the transducer. The data will be downloaded during the routine visits to the Site. A corrosion-resistant stream gage consisting of an iron frame coated with baked enamel (such as available from Ben Meadows) will be pounded into the substrate in approximately 5 feet of water. Then, perforated polyvinyl chloride (PVC) section will be attached to it and the transducer will be inserted into the PVC. The top of stake will be surveyed and the distance from transducer to top of stake will be measured to calculate the transducer elevation. Finally, the field crew will paddle over to the stake to download the data from the data logger.

Additional surface water samples are not planned; however, opportunistic samples will be collected if something unusual is observed while routine field monitoring, such as if the pit is off-color or an unusual pH is measured. Water samples for laboratory analyses from the pit lake will be collected in the area of the observed unusual (chemistry) pattern. Parameters to be analyzed are provided in Table 3-2. This will require accessing the monitoring platform by boat and collecting the sample from the selected depth using a peristaltic pump. In addition to the water sample, a sonde and data logger equipped with calibrated probes for pH, specific conductance, temperature, ORP, turbidity, and DO will be lowered vertically through the water column of the pit lake. Readings will be taken every five feet and recorded.

Analytical results for all samples will be validated following procedures specified in the *Site-wide QAPP* (Volume 1 of Black & Veatch, 2011). Results will be entered into the project database.

**Requirements:** Pit lake samples will be collected as described above. Required analyses are summarized in Table 3-2. Sample volumes, container requirements, preservation, and handling are shown in Table 3-3. One filter rinse blank per lot of filters will be required prior to collection of surface water samples for TAL metals. The number of rinse blanks shown in Table 3-2 assumes that the same lot of filters will be used during collection of surface water samples. Surface water samples will be shipped to SVL, Inc. in Kellogg, ID for analysis (see Table 3-2 for shipping address). All new sample stations will be located with a GPS unit per the SESD SOP.

EPA-SESD SOPs relevant to surface water sampling include:

- SESDPROC-100-R3 Field pH Measurement
- SESDPROC-101-R5 Field Specific Conductance Measurement
- SESDPROC-102-R3 Field Temperature Measurement
- SESDPROC-103-R3 Field Turbidity Measurement
- SESDPROC-106-R2 Field Measurement of Dissolved Oxygen
- SESDPROC-108-R4 Equipment Inventory and Management
- SESDPROC-110-R3 Global Positioning System
- SESDPROC-113-R1 Field Measurement of ORP
- SESDPROC-201-R3 Surface Water Sampling
- SESDPROC-203-R3 Pump Operation
- SESDPROC-005-R2 Sample and Evidence Management
- SESDPROC-010-R5 Logbooks
- SESDPROC-011-R4 Field Sampling Quality Control
- SESDPROC-205-R2 Field Equipment Cleaning and Decontamination
- SESDPROC-209-R2 Packing, Marking, Labeling and Shipping of Environmental and Waste Samples

### **3.2 Storm Water Runoff Sampling – Two Rain Storm Events**

In addition to the stream sampling discussed above, 6 surface runoff samples (per storm event) will be collected from select locations to determine water quality entering the pit lake and leaving the Site.

**Objective:** To determine the potential contribution of runoff water quality that may impact the pit lake. In addition, runoff water quality leaving the Site in eroded washes and/or overflow from process ponds will be assessed.

**Approach:** Grab samples of surface water will be used to provide field and analytical data for these locations. Parameters to be analyzed for are provided in Table 3-2. Locations are shown on Figure 3-1.

It is anticipated that samples in washes will be collected either by direct filling of bottles, or using a stainless steel scoop to transfer water to bottles. A peristaltic pump with Teflon® tubing may be used to collect water and fill bottles from the pump outlet.

Crude estimates of discharge will be made, limited to site descriptions, estimations of wash dimensions, and simple time of travel measurements.

An attempt will be made to collect runoff from the high-walls on the east side of the mine pit. A small boat will be used to approach the wall. Clean, stainless steel scoops or bowls may be used to collect water running down the high-wall at two general locations. Total and dissolved metals will be the preferential analyses if collected volume is limited. Lightning or difficulty in collecting adequate volume may interfere with the collection of these samples.

In order to collect samples quickly during the rain event, a sample container for dissolved metals will be filled directly at the sampling site. The sample will be filtered later, using a peristaltic pump and a 0.45 micrometre ( $\mu\text{m}$ ) filter. Several hours may elapse prior to filtering.

As in the collection of dissolved metals samples, a container will be filled for the measurement of field parameters after the fact. Specific Conductance, pH, and turbidity will be measured and recorded. The relevant SESD SOPs listed in Section 3.1 will be followed.

### **3.3 Storm Water Sampling in Creek Tributaries – Two Rain Storm Events**

SESD prepared a QAPP in April 2013 titled “*Barite Hills Remedial Investigation, Storm Event Sampling*” to guide the collection of first flush samples from a rain event. On May 4, 2013, a storm event triggered the collection of Isco samplers at two of three locations at the Site. The magnitude of the rainfall at that time was approximately two inches over a 24 hour period. The purpose of continuing the storm water sampling at the same locations in the SESD QAPP is to determine water quantity (stream discharge measurements) and quality of water that may impact downstream wetlands.

When a sizeable rain event greater than 2 inches within a 24 hour period is expected, Black & Veatch will mobilize to retrieve samples for analysis.

In addition to the analytical samples, measurements of stream discharge will be made at three locations so contaminant loads from the site can be calculated for the Hawe Creek tributaries. Stream discharge measurements were made during the May 2011 surface water sampling event at base flow conditions.

**Objective:** To obtain information on the quantity and quality of storm water in the tributary streams of Hawe Creek and to determine contaminant loads.

**Approach:** Use Isco brand samplers to provide periodic samples throughout a runoff event. Temporary flow measurement devices, such as weirs, will be installed at the following locations to measure discharge under runoff conditions:

- BH118 in north tributary upstream of the Main Pit spillway;
- BH119 in north tributary downstream of the Main Pit spillway;
- BH136 just upstream of BH129 in the flowing portion of the west tributary upstream of the west beaver pond.

Locations will be established prior to final field setup and will be based on the previous SESD QAPP (EPA, 2013) and the May 2013 results. Together, the discharge and water quality measurements will be used to compute changes in contaminant mass load throughout a runoff event and to characterize the magnitude of any first flush signatures that might be observed.

**Requirements:** Surface water samples will be collected during storm runoff conditions. The SESD QAPP identified sampling triggers and Isco sampling intervals for each of the three stations listed above. Based on the results of the May 4, 2013 storm event, the following sampling triggers and Isco intervals would be needed:

Sampling Triggers		
Station	Stage (ft)	Rate of Change (ft/hr)
BH118	0.40	0.65
BH119	1.28	1.47
BH136	1.13	4.15

Isco Sampling Intervals			
Station	Sampling Interval (min)	Sampling Time (hr)	Time to Peak (hr)
BH118	5	1.0	0.88
BH119	8	1.6	1.63
BH136	5	1.0	0.87

Weirs or other appropriate temporary flow structures will be installed at the three locations. Criteria used to install these devices will be similar to those outlined in *Performing Quality Flow Measurements at Mine Sites*, EPA/600/R-01/43. Pressure transducers with data loggers will be installed behind the weir to provide a continuous recording of stream stage that can be converted into stream discharge



using the appropriate weir equation. Isco samplers will be placed at each of the three weirs. Table 3-4 lists the parameters for the high flow Isco sampling (storm event sampling) and Table 3-5 shows the methods and minimum containers. One filter rinse blank per lot of filters will be required prior to collection of surface water samples for TAL metals.

## 4.0 References

Black & Veatch, 2010. Black & Veatch Special Projects Corp. *Remedial Investigations / Feasibility Study Work Plan for Barite Hill Goldfields Site*. August, 2010.

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EPA, 2011f. U.S. Environmental Protection Agency Region 4, Science and Ecosystem Support Division (SESD). Barite Hill Remedial Investigation, Potable Well Sampling Quality Assurance Project Plan. SESD Project # 11-0183, September 9, 2011.

EPA, 2013. U.S. Environmental Protection Agency Region 4, Science and Ecosystem Support Division (SESD). Barite Hill Remedial Investigation, Storm Event Sampling Quality Assurance Project and Study Plan. SESD Project # 13-0262, April 18, 2013.

## TABLES



**Table 3-1**  
**Monitoring Overview – Barite Hill Pit Lake and Diversion Channels**  
**Barite Hill/Nevada Goldfields Site**  
**McCormick County, South Carolina**

Parameter	Method	Duration	Frequency	Rationale
<b>Routine Monitoring</b>				
Water elevation	Pressure transducer with data logger	1 year, then continue as post-RI monitoring	Daily from 1 location	Understanding water level in pit with respect to rain and drought events (dilution and evapoconcentration); potential flushing of submerged dump as level fluctuates.
Bathymetry	Grid pattern using boat-mounted depth sounder	Single measurement	Once	Provide data that will allow calculation of the quantity of water in the pit lake that can be tied to measured pit lake elevation.
Total organic carbon	Analytical	1 year, then continue as post-RI monitoring	Monthly from at least 4 depths	Information on carbon concentrations over time that support continued bacterial action which removes metals.
Ferrous and total iron	Field test kit	1 year, then continue as post-RI monitoring	Monthly from at least 4 depths	Information on ferric iron concentrations over time to ensure that the mackinawite cap remains intact.
Total alkalinity	Analytical	1 year, then continue as post-RI monitoring	Monthly from at least 4 depths	Information on alkalinity concentrations over time that support continued buffering of acid additions to the pit lake.
Field parameters (monthly)	Hand-drop multiprobe sonde	1 year, then continue as post-RI monitoring	Monthly at 5 foot intervals from at least one location	Provide information on temperature, pH, specific conductance, dissolved oxygen, redox potential, and turbidity through the pit water column that supplements data collected from the automatic system.

**Table 3-1**  
**Monitoring Overview – Barite Hill Pit Lake and Diversion Channels**  
**Barite Hill/Nevada Goldfields Site**  
**McCormick County, South Carolina**

Parameter	Method	Duration	Frequency	Rationale
<b>Opportunistic Samples (Two Rain Storm Events)</b>				
Field parameters (opportunistic)	Hand-drop multiprobe sonde	As needed to capture perturbing events such as major rainfall or drought	At 5 foot intervals from at least one location	Provide information on temperature, pH, specific conductance, dissolved oxygen, redox potential, and turbidity through the pit water column that supplements data collected from the automatic system.
Rainfall pH	Field meter	At onset of rain (when enough water can be collected), then about 30 minutes after continuous rain	2 storms	Provide some understanding of proton ( $H^+$ ) acidity added to the pit by rainfall events.
Ferrous and total iron	Field test kit	As needed to capture perturbing events such as major rainfall or drought	At least 4 depths	Information on ferric iron concentrations over time to ensure that the mackinawite cap remains intact.
Total alkalinity	Analytical	As needed to capture perturbing events such as major rainfall or drought	At least 4 depths	Information on alkalinity concentrations over time that support continued buffering of acid additions to the pit lake.
Pit wall runoff samples	Lab analysis & field parameters	During rainfall runoff from pit wall	Two locations	Info on runoff quality and mass load to pit lake. Lab should include metals, sulfate, chloride, acidity, nitrates; field parameters should include pH, specific conductance, temperature, and redox potential.
Storm water diversion samples	Lab analysis, field parameters, & discharge	During rainfall runoff event	Two storms of different sizes	Info on runoff quality and mass load to pit lake. Lab should include metals, sulfate, chloride, acidity, nitrates; field parameters include pH, specific conductance, temperature, and redox potential. Discharge should be measured throughout the runoff event so that a volume of water entering the pit can be computed for each channel.

**Table 3-2**  
**Surface Water Parameters**  
**Barite Hill/Nevada Goldfields Site**  
**McCormick County, South Carolina**

Station ID	Media	Sample Type	Total Metals	Filtered Metals	Anions	Total Acidity	Total Alkalinity	TDS & TSS	TOC	Field Parameter	Approximate Coordinates	
											Easting	Northing
Pit Lake Water Samples												
BH101	SW	Discrete					X		X	X <sup>#</sup>	off pit lake raft *	
BH104	SW	Discrete					X		X	X <sup>#</sup>	33.8754977	-82.2935497
TBD <sup>+</sup>	SW	Discrete	X	X	X	X	X	X	X	X <sup>#</sup>	off pit lake raft *	
Surface Water Runoff Samples												
BH152	SW	Discrete	X	X	X	X	X	X	X	X <sup>#</sup>	Downgradient reach of ravine south of reuseable leach pads	
BH153	SW	Discrete	X	X	X	X	X	X	X	X <sup>#</sup>	Capture of runoff below seep at permanent leach pile	
BH154	SW	Discrete	X	X	X	X	X	X	X	X <sup>#</sup>	Runoff from graded area	
BH155	SW	Discrete	X	X	X	X	X	X	X	X <sup>#</sup>	Runoff from graded area, south end of pond	
BH156	SW	Discrete	X	X	X	X	X	X	X	X <sup>#</sup>	Mine pit high-wall sample, exposed rock area	
BH157	SW	Discrete	X	X	X	X	X	X	X	X <sup>#</sup>	Mine pit high-wall sample, weathered soil area	

**Notes:**

\* Deep water sample @ 50 ft depth

+ Sample if needed per unforeseen circumstance, see Field Sampling Plan

^ 3-point composite

# every 5 ft depth

ORP - oxidation reduction potential

pH - hydrogen ion concentration

TBD - To be determined

TDS - Total dissolved solids

TOC - Total organic carbon

TSS - Total suspended solids

**Sample shipping:**

**Analytical samples**

Chris Meyer

SVL, Inc.

One Government Gulch

Kellogg, ID 83837

800-597-7144

**Total and Dissolved Metals**

Aluminum	Magnesium
Antimony	Manganese
Arsenic	Molybdenum
Barium	Nickel
Beryllium	Potassium
Cadmium	Selenium
Calcium	Silver
Chromium	Sodium
Cobalt	Thallium
Copper	Vanadium
Iron	Zinc
Lead	

**Anions**

Sulfate  
Chloride

**Others**

TDS  
TSS  
Total Acidity  
Total Alkalinity  
TOC

**Field Parameters**

pH  
Specific Conductivity  
Temperature  
ORP  
Turbidity  
Total Fe  
Ferrous Fe  
Dissolved Oxygen



**Table 3-3**  
**Surface Water Samples**  
**Barite Hill/Nevada Goldfields Site**  
**McCormick County, South Carolina**

Analysis	Analytical Method	Container Type	Preservative	Holding Time	Samples*	QC Samples <sup>2, 3</sup>				Total Numbers of Samples	Total Numbers of Containers
						Equipment Rinsate	Trip Blank	Field Duplicates	MS/MSD		
Pit Lake Water Samples - Analytical - PER STORM EVENT											
TOC	EPA SW-846 (Method 9065)	1 x 500 mL poly	Ice to 4oC	7 days	5	0	0	0	0	5	5
Alkalinity	EPA Method 310.1	1 x 500 mL poly	Ice to 4°C	14 days	5	0	0	0	0	5	5
TAL Metals (Unfiltered)	EPA SW-846 (Method 6010B or 6020 series)	1 x 1 L poly	HNO <sub>3</sub> (pH<2), Ice to 4°C	180 days	1	0	0	0	0	1	1
TAL Metals (Filtered)	EPA SW-846 (Method 6010B or 6020 series)	1 x 1 L poly	HNO <sub>3</sub> (pH<2), Ice to 4°C	180 days	1	1	0	0	0	2	2
Anions	EPA Method 300.0	1 x 500 mL poly	Ice to 4°C	28 days	1	0	0	0	0	1	1
Acidity	EPA Method 305.1	1 x 500 mL poly	Ice to 4°C	14 days	1	0	0	0	0	1	1
TDS/TSS	EPA Methods 160.1, 160.2	1 x 1 L poly	Ice to 4°C	7 days	1	0	0	0	0	1	1
Surface Water Runoff Samples - Analytical - PER STORM EVENT											
TOC	EPA SW-846 (Method 9065)	1 x 500 mL poly	Ice to 4oC	7 days	6	0	0	1	1	8	8
TAL Metals (Unfiltered)	EPA SW-846 (Method 6010B or 6020 series)	1 x 1 L poly	HNO <sub>3</sub> (pH<2), Ice to 4°C	180 days	6	0	0	1	1	8	8
TAL Metals (Filtered)	EPA SW-846 (Method 6010B or 6020 series)	1 x 1 L poly	HNO <sub>3</sub> (pH<2), Ice to 4°C	180 days	6	1	0	1	1	9	9
Anions	EPA Method 300.0	1 x 500 mL poly	Ice to 4°C	28 days	6	0	0	1	1	8	8
Acidity	EPA Method 305.1	1 x 500 mL poly	Ice to 4°C	14 days	6	0	0	1	1	8	8
Alkalinity	EPA Method 310.1	1 x 500 mL poly	Ice to 4°C	14 days	6	0	0	1	1	8	8
TDS/TSS	EPA Methods 160.1, 160.2	1 x 1 L poly	Ice to 4°C	7 days	6	0	0	1	1	8	8

**Notes:**

\* - Actual number of samples To Be Determined.

<sup>1</sup> - Pre-preserved bottles usually have insufficient NaOH to keep pH>12 due to very acidic water. Field crew should keep extra NaOH pellets.

<sup>2</sup> - a temperature blank will be included in each cooler shipped.

<sup>3</sup> - a preservative blank will be included per shipment.

°C - degrees Celsius

ASTM - American Society for Testing and Materials

EPA - U.S. Environmental Protection Agency

mL - milliliters

MS - Matrix Spike

MSD - Matrix Spike Duplicate

HNO<sub>3</sub> - Nitric Acid

QC - quality control

TAL - Target Analyte List

TDS - Total dissolved solids

TOC - Total Organic Carbon

TSS - Total suspended solids

**Table 3-4**  
**Rain Event Surface Water Parameters**  
**Barite Hill/Nevada Goldfields Site**  
**McCormick County, South Carolina**

Station	Media	Sample Type	Total Metals	Filtered Metals	Anions	Total Acidity	Total Alkalinity	TDS & TSS	Field Parameter	Location		
										Lat	Lon	Field location
Isco Samples												
BH118	SW	Discrete	X	X	X	X	X	X	X	33.877567	-82.295124	north tributary
BH119	SW	Discrete	X	X	X	X	X	X	X	33.876447	-82.304775	north tributary
BH136	SW	Discrete	X	X	X	X	X	X	X	33.870128	-82.321684	south tributary

**Notes:**

pH - hydrogen ion concentration

TSS - Total suspended solids

TDS - Total dissolved solids

eH/ORP - Redox potential / oxidation reduction potential

**Total and Dissolved Metals**

Aluminum	Magnesium
Antimony	Manganese
Arsenic	Molybdenum
Barium	Nickel
Beryllium	Potassium
Cadmium	Selenium
Calcium	Silver
Chromium	Sodium
Cobalt	Thallium
Copper	Vanadium
Iron	Zinc
Lead	

**Anions**

Sulfate  
Chloride

**Others**

TDS  
TSS  
Total Acidity  
Total Alkalinity

**Field Parameters**

pH  
Specific Conductivity  
Temperature  
Eh/ORP  
Turbidity  
Dissolved Oxygen



**Table 3-5**  
**Surface Water Samples for Rain Event Sampling**  
**Barite Hill/Nevada Goldfields Site**  
**McCormick County, South Carolina**

Analysis	Analytical Method	Container Type	Preservative	Holding Time	Samples	QC Samples <sup>1, 2</sup>				Total Numbers of Samples	Total Numbers of Containers
						Equipment Rinsate	Trip Blank	Field Duplicates	MS/MSD		
Surface Water (Isco) Samples for High Flow Sampling - PER STORM EVENT											
TAL Metals (Filtered)	EPA SW-846 (Method 6010B or 6020 series)	1 x 1 L poly	HNO <sub>3</sub> (pH<2), Ice to 4°C	180 days	3	1	0	1	1	6	6
Anions	EPA Method 300.0	1 x 500 mL poly	Ice to 4°C	28 days	3	0	0	1	0	4	4
Acidity	EPA Method 305.1	1 x 500 mL poly	Ice to 4°C	14 days	3	0	0	1	0	4	4
Alkalinity	EPA Method 310.1	1 x 500 mL poly	Ice to 4°C	14 days	3	0	0	1	0	4	4
TDS/TSS	EPA Methods 160.1, 160.2	1 x 1 L poly	Ice to 4°C	7 days	3	0	0	1	0	4	4

**Notes:**

<sup>1</sup> - a temperature blank will be included in each cooler shipped.

<sup>2</sup> - a preservative blank will be included per shipment.

°C - degrees Celsius

EPA - U.S. Environmental Protection Agency

MS - Matrix Spike

MSD - Matrix Spike Duplicate

pH - hydrogen ion concentration

QC - quality control

TAL - Target Analyte List

TDS - Total dissolved solids

TSS - Total suspended solids

**FIGURE**





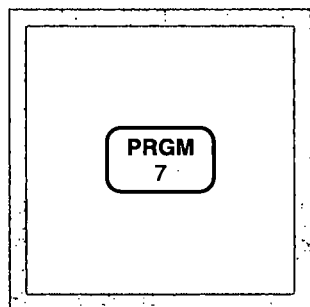


**Attachment 1**  
**Iron Test Kit – Standard Operating Procedure**

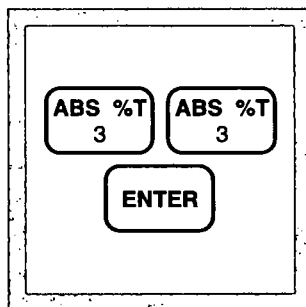


**IRON, FERROUS (0 to 3.00 mg/L)**

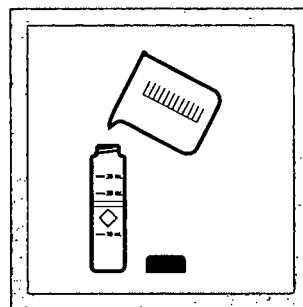
For water, wastewater, and seawater

**1,10 Phenanthroline Method\* (Powder Pillows or AccuVac Ampuls)****Using Powder Pillows**

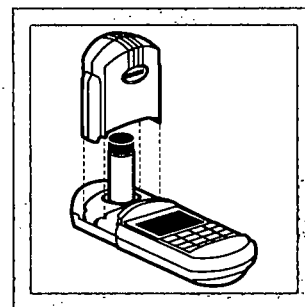
1. Enter the stored program number for Ferrous iron ( $\text{Fe}^{2+}$ )-powder pillows.



2. Press: **33 ENTER**  
The display will show **mg/L, Fe** and the **ZERO** icon.



3. Fill a sample cell with 25 mL of sample (the blank).



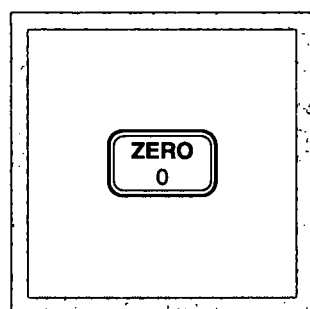
4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

Press: **PRGM**

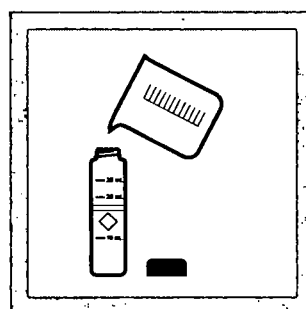
The display will show:

**PRGM ?**

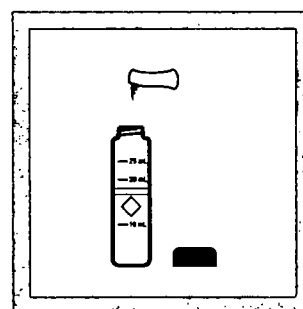
*Note: Analyze samples as soon as possible to prevent oxidation of ferrous iron to ferric iron, which is not determined.*



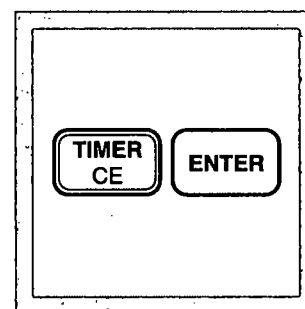
5. Press: **ZERO**  
The cursor will move to the right, then the display will show:  
**0.00 mg/L Fe**



6. Fill another sample cell with 25 mL of sample.



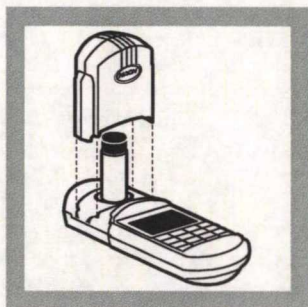
7. Add the contents of one Ferrous Iron Reagent Powder Pillow to the sample cell (the prepared sample). Cap and invert to mix.  
*Note: Undissolved powder does not affect accuracy.*



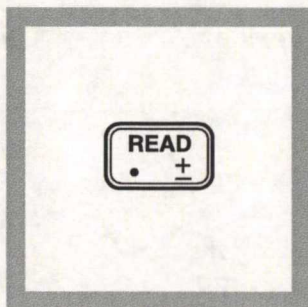
8. Press:  
**TIMER ENTER**  
A three-minute reaction period will begin.  
*Note: An orange color will form if ferrous iron is present.*

\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

## IRON, FERROUS, continued



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L ferrous iron will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

### Using AccuVac Ampuls



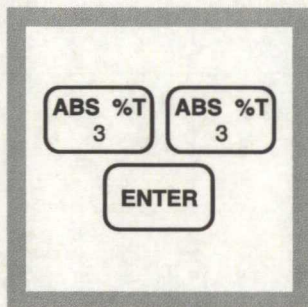
1. Enter the stored program number for ferrous iron ( $\text{Fe}^{2+}$ ) AccuVac ampuls.

Press: **PRGM**

The display will show:

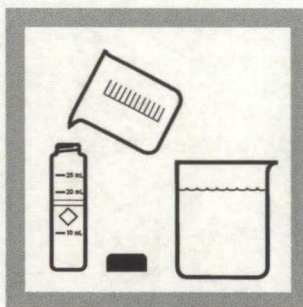
**PRGM ?**

*Note: Analyze samples as soon as possible to prevent air oxidation of ferrous iron to ferric, which is not determined.*

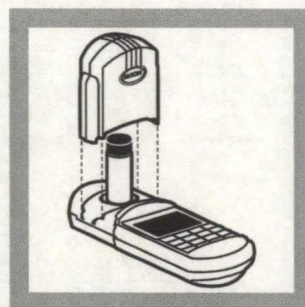


2. Press: **33 ENTER**

The display will show **mg/L, Fe** and the **ZERO** icon.

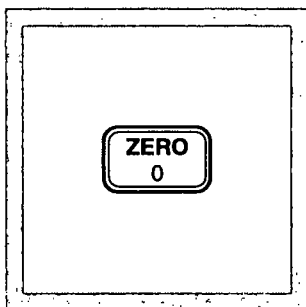


3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

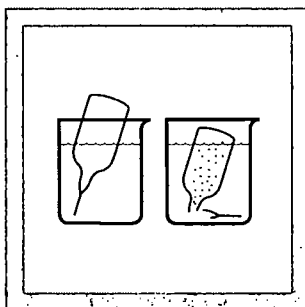
## IRON, FERROUS, continued



**5. Press: ZERO**

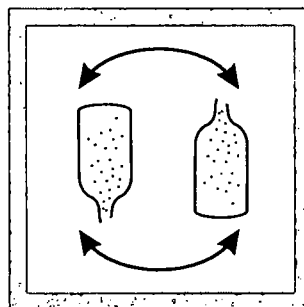
The cursor will move to the right, then the display will show:

**0.00 mg/L Fe**



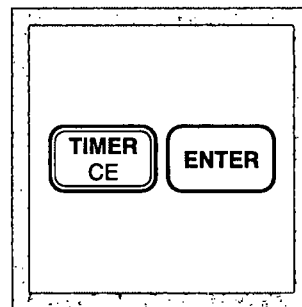
**6. Fill a Ferrous Iron AccuVac Ampul with sample.**

*Note: Keep the tip immersed while the ampul fills completely.*



**7. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.**

*Note: Undissolved powder does not affect accuracy.*

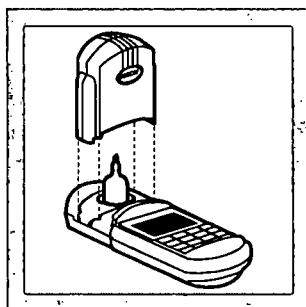


**8. Press:**

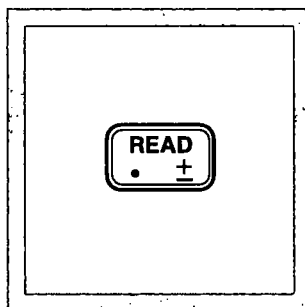
**TIMER ENTER**

A three-minute reaction period will begin.

*Note: An orange color will form if ferrous iron is present.*



**9. Place the AccuVac ampul into the cell holder. Tightly cover the sample cell with the instrument cap.**



**10. Press: READ**

The cursor will move to the right, then the result in mg/L ferrous iron will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*



## IRON, FERROUS, continued

---

### Sampling and Storage

Ferrous iron must be analyzed immediately and cannot be stored. Analyze samples as soon as possible to prevent oxidation of ferrous iron to ferric iron, which is not measured.

### Accuracy Check

#### Standard Solution Method

Prepare a ferrous iron stock solution (100 mg/L  $\text{Fe}^{2+}$ ) by dissolving 0.7022 grams of ferrous ammonium sulfate, hexahydrate, in deionized water. Dilute to 1 liter. Prepare immediately before use. Dilute 1.00 mL of this solution to 100 mL with deionized water to make a 1.00 mg/L standard solution. Prepare immediately before use.

Run the test using the 1.00 mg/L  $\text{Fe}^{2+}$  Standard Solution by following either the powder pillow or AccuVac procedure. Results should be between 0.90 mg/L and 1.10 mg/L  $\text{Fe}^{2+}$ .

### Method Performance

#### Precision

In a single laboratory using an iron standard solution of 2.00 mg/L  $\text{Fe}^{2+}$  and two representative lots of powder pillow reagents with the instrument, a single operator obtained a standard deviation of  $\pm 0.017$  mg/L  $\text{Fe}^{2+}$ .

In a single laboratory using a standard solution of 2.00 mg/L  $\text{Fe}^{2+}$  and two representative lots of AccuVac ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 0.009$  mg/L  $\text{Fe}^{2+}$ .

#### Estimated Detection Limit

The estimated detection limit for program 33 (powder pillows and AccuVac Ampuls) is 0.03 mg/L Fe. For more information on the estimated detection limit, see *Section 1*.

### Summary of Method

The 1,10-phenanthroline indicator in Ferrous Iron Reagent reacts with ferrous iron in the sample to form an orange color in proportion to the iron concentration. Ferric iron does not react. The ferric iron ( $\text{Fe}^{3+}$ ) concentration can be determined by subtracting the ferrous iron concentration from the results of a total iron test.



## IRON, FERROUS, continued

### REQUIRED REAGENTS & APPARATUS (USING POWDER PILLOWS)

Description	Quantity Required		Units	Cat. No.
	Per Test			
Ferrous Iron Reagent Powder Pillows.....	1 pillow.....	100/pkg	.....	1037-69
Sample Cell, 10-20-25 mL, w/ cap .....	2 .....	6/pkg	.....	24019-06

### REQUIRED REAGENTS & APPARATUS (USING ACCUVAC AMPULS)

Ferrous Iron Reagent AccuVac Ampuls.....	1 ampul.....	25/pkg	.....	25140-25
Beaker, 50 mL .....	1 .....	each	.....	500-41H

### OPTIONAL REAGENTS

Ferrous Ammonium Sulfate, hexahydrate, ACS.....	113 g.....	11256-14
Water, deionized .....	4 L .....	272-56

### OPTIONAL APPARATUS

AccuVac Snapper Kit .....	each	.....	24052-00
Balance, analytical, 115 V, 0.1 mg .....	each	.....	28014-01
Balance, analytical, 230 V, 0.1 mg .....	each	.....	28014-02
Clippers, for opening powder pillows .....	each	.....	968-00
Flask, volumetric, 100 mL, Class A.....	each	.....	14574-42
Flask, volumetric, 1000 mL, Class A.....	each	.....	14574-53
Pipet, volumetric, Class A, 1.00 mL .....	each	.....	14515-35
Pipet Filler, safety bulb .....	each	.....	14651-00
Weighing Boat, 67/46 mm, 8.9 cm square .....	500/pkg	.....	21790-00

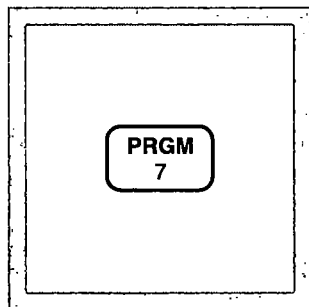
### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

**IRON, TOTAL (0 to 3.00 mg/L)**

For water, wastewater, and seawater

**FerroVer Method (Powder Pillows or AccuVac Ampuls)****USEPA approved for reporting wastewater analysis (digestion is required; see Section 2\*)**

**1.** Enter the stored program number for iron (Fe) powder pillows.

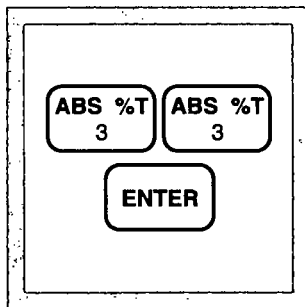
Press: **PRGM**

The display will show:

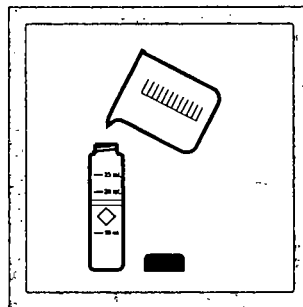
**PRGM ?**

*Note: Determination of total iron requires a digestion prior to analysis (see Section 2).*

*Note: Adjust pH of stored samples before analysis.*

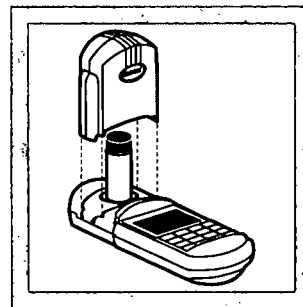


**2.** Press: **33 ENTER**  
The display will show **mg/L, Fe** and the **ZERO** icon.

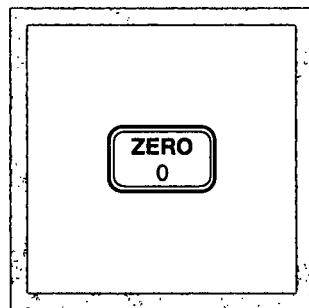


**3.** Fill a clean sample cell with 10 mL of sample (the blank).

*Note: For turbid samples, treat the blank with one 0.1-gram scoop of RoVer Rust Remover. Swirl to mix.*

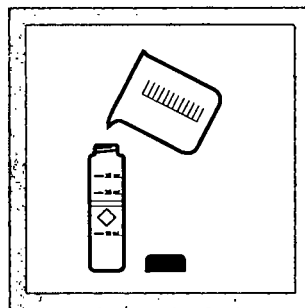


**4.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

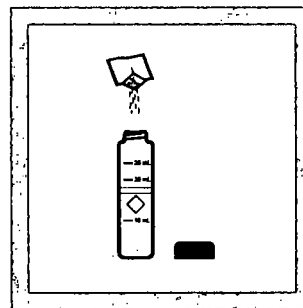


**5.** Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0.00 mg/L Fe**

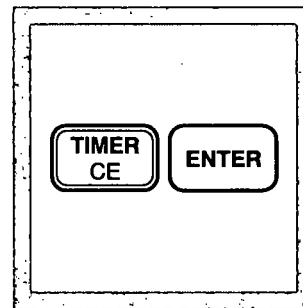


**6.** Fill another sample cell with 10 mL of sample.



**7.** Add the contents of one FerroVer Iron Reagent Powder Pillow to the sample cell (the prepared sample). Cap and invert to dissolve the reagent powder.

*Note: Accuracy is not affected by undissolved powder.*



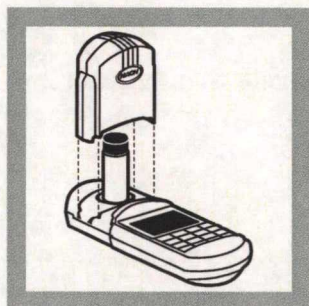
**8.** Press: **TIMER ENTER**  
A three-minute reaction period will begin.

*Note: An orange color will form if iron is present.*

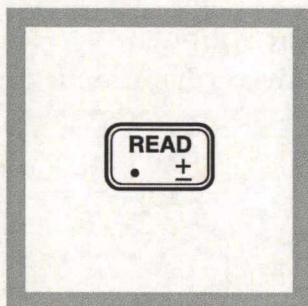
*Note: Samples containing visible rust should be allowed to react at least five minutes.*

\* Federal Register, 45 (126) 43459 (June 27, 1980). See also 40 CFR, part 136.3, Table IB.

## IRON, TOTAL, continued



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

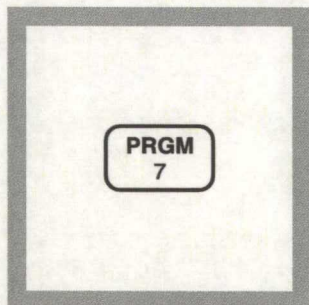


10. Press: **READ**

The cursor will move to the right, then the result in mg/L iron (Fe) will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

### Using AccuVac Ampuls



1. Enter the stored program number for iron (Fe), AccuVac ampuls.

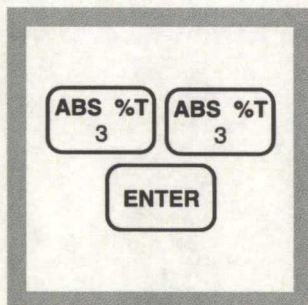
Press: **PRGM**

The display will show:

**PRGM ?**

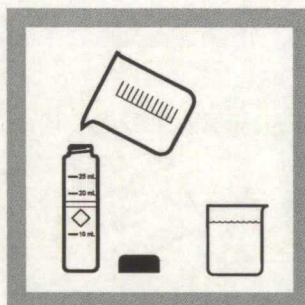
*Note: Adjust pH of stored samples before analysis.*

*Note: Determination of total iron requires a digestion prior to analysis (see Section 2).*



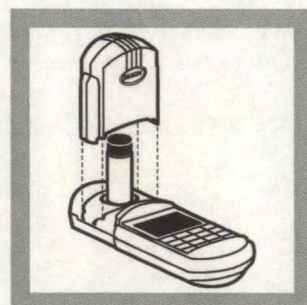
2. Press: **33 ENTER**

The display will show **mg/L, Fe** and the **ZERO** icon.



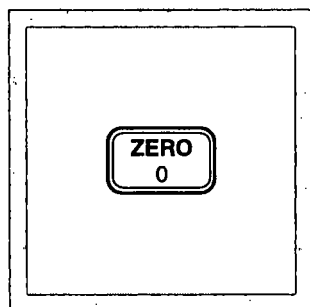
3. Fill a sample cell (the blank) with at least 10 mL of sample. Collect at least 40 mL of sample in a 50-mL beaker.

*Note: For turbid samples, treat the blank with one 0.1 g scoop of RoVer Rust Remover. Swirl to mix.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

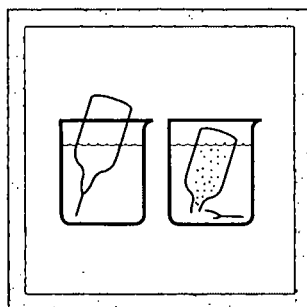
## IRON, TOTAL, continued



**5. Press: ZERO**

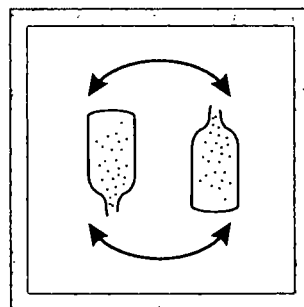
The cursor will move to the right, then the display will show:

**0.00 mg/L Fe**



**6. Fill a FerroVer AccuVac Ampul with sample.**

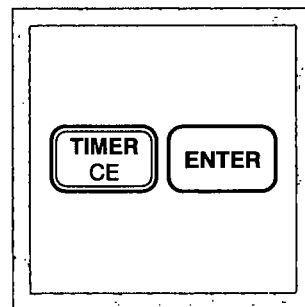
*Note: Keep the tip immersed while the ampul fills completely.*



**7. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.**

*Note: An orange color will form if iron is present.*

*Note: Accuracy is not affected by undissolved powder.*

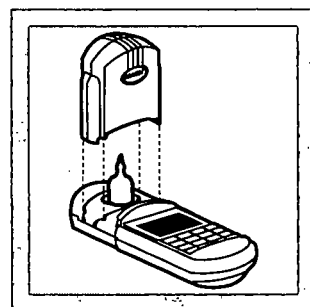


**8. Press:**

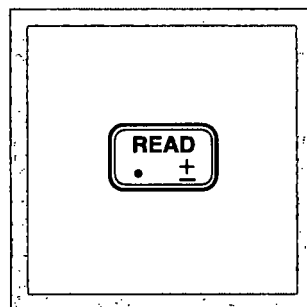
**TIMER ENTER**

A three-minute reaction period will begin.

*Note: Samples containing visible rust should be allowed to react at least five minutes.*



**9. Place the AccuVac ampul into the cell holder. Tightly cover the ampul with the instrument cap.**



**10. Press: READ**

The cursor will move to the right, then the result in mg/L iron (Fe) will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*



## IRON, TOTAL, continued

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### Sampling and Storage

Collect samples in acid-cleaned glass or plastic containers. No acid addition is necessary if analyzing the sample immediately. To preserve samples, adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. Adjust the pH to between 3 and 5 with 5.0 N Sodium Hydroxide Standard Solution before analysis. Correct the test result for volume additions; see *Correcting for Volume Additions* in *Section 1* for more information. If only dissolved iron is to be determined, filter the sample before adding the acid.

### Accuracy Check

#### Standard Additions Method

- a) Snap the neck off a 50 mg/L Iron PourRite Ampule Standard Solution.
- b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to three 25-mL samples and mix thoroughly.
- c) For analysis using AccuVac Ampuls, transfer solutions to dry, clean 50-mL beakers to facilitate filling of the ampuls. For analysis with powder pillows, transfer only 10 mL of solution to the 10-mL sample cells.
- d) Analyze each standard addition sample as described above. The iron concentration should increase 0.2 mg/L for each 0.1 mL of standard added.
- e) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

#### Standard Solution Method

Prepare a 1.0-mg/L iron standard by diluting 1.00 mL of Iron Standard Solution, 100 mg/L Fe, to 100 mL with deionized water. Or, dilute 1.00 mL of an Iron PourRite Ampule Standard Solution (50 mg/L) to 50 mL in a volumetric flask. Prepare this solution daily.

Run the test following the procedure for powder pillows or AccuVac Ampuls. Results should be between 0.90 mg/L and 1.10 mg/L Fe.

## IRON, TOTAL, continued

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 2.00 mg/L Fe and two representative lots of powder pillow reagents with the instrument, a single operator obtained a standard deviation of  $\pm 0.017$  mg/L.

In a single laboratory, using a standard solution of 2.00 mg/L Fe and two representative lots of AccuVac ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 0.009$  mg/L Fe.

#### Estimated Detection Limit (EDL)

The EDL for program 33 is 0.03 mg/L Fe. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

### Interferences

#### Interfering Substances and Suggested Treatments

Interfering Substance	Interference Level and Treatment
Calcium, $\text{Ca}^{2+}$	No effect at less than 10,000 mg/L as $\text{CaCO}_3$
Chloride, $\text{Cl}^-$	No effect at less than 185,000 mg/L.
Copper, $\text{Cu}^{2+}$	No effect. Masking agent is contained in FerroVer Iron Reagent.
High Iron Levels	Inhibits color development. Dilute sample and retest to verify results.
Iron Oxide	Requires mild, vigorous or Digesdahl digestion (see Section 2). After digestion, adjust sample to pH 3-5 with sodium hydroxide, then analyze.
Magnesium	No effect at 100,000 mg/L as $\text{CaCO}_3$ .
Molybdate, Molybdenum	No effect at 25 mg/L as Mo.
High Sulfide Levels, $\text{S}^{2-}$	<ol style="list-style-type: none"><li>1. Treat in fume hood or well-ventilated area. Add 5 mL HCl to 100 mL sample in a 250-mL Erlenmeyer flask. Boil 20 minutes.</li><li>2. Cool. Adjust pH to 3-5 with NaOH. Re-adjust volume to 100 mL with deionized water.</li><li>3. Analyze.</li></ol>



## IRON, TOTAL, continued

Interfering Substance	Interference Level and Treatment
Turbidity	<ol style="list-style-type: none"> <li>1. Add 0.1 g scoop of RoVer Rust Remover to the blank in Step 3. Swirl to mix.</li> <li>2. Zero the instrument with this blank.</li> <li>3. If sample remains turbid, add three 0.2 g scoops of RoVer to a 75-mL sample. Let stand 5 minutes.</li> <li>4. Filter through a glass filter or centrifuge.</li> <li>5. Use filtered sample in Steps 3 and 6.</li> </ol>
Sample pH (extreme)	Adjust pH to 3-5. See <i>Interferences</i> in Section 1.
Highly Buffered Samples	Adjust pH to 3-5. See <i>Interferences</i> in Section 1.

### Summary of Method

FerroVer Iron Reagent reacts with all soluble iron and most insoluble forms of iron in the sample to produce soluble ferrous iron. This reacts with 1,10-phenanthroline indicator in the reagent to form an orange color in proportion to the iron concentration.

### REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required		Cat No.
	Per Test	Unit	
FerroVer Iron Reagent Powder Pillows .....	1 pillow .....	100/pkg.....	21057-69
Sample cell, 10-20-25 mL, with screw cap .....	1 .....	6/pkg.....	24019-06

### REQUIRED REAGENTS & APPARATUS (Using AccuVac Ampuls)

FerroVer Iron Reagent AccuVac Ampuls .....	1 ampul .....	25/pkg.....	25070-25
Beaker, 50 mL .....	1 .....	each.....	500-41H

### OPTIONAL REAGENTS

Description	Unit	Cat. No.
Ammonium Hydroxide, ACS .....	500 mL.....	106-49
Drinking Water Standard, Metals, LR (Cu, Fe, Mn) .....	500 mL.....	28337-49
Drinking Water Standard, Metals, HR (Cu, Fe, Mn) .....	500 mL.....	28336-49
Hydrochloric Acid Standard Solution, 6 N.....	500 mL.....	884-49
Hydrochloric Acid, ACS.....	500 mL.....	134-49
Iron Standard Solution, 100 mg/L .....	100 mL.....	14175-42
Iron Ampule Standard, 50 mg/L .....	20/pkg.....	14254-20
Nitric Acid, ACS.....	500 mL.....	152-49
Nitric Acid Solution, 1:1.....	500 mL.....	2540-49
RoVer Rust Remover .....	454 g.....	300-01
Sodium Hydroxide Standard Solution, 5.0 N .....	100 mL MDB.....	2450-32
Water, deionized.....	4 L.....	272-56



## IRON, TOTAL, continued

### OPTIONAL APPARATUS

AccuVac Snapper Kit .....	each .....	24052-00
Ampule Breaker, PourRite Ampules .....	each .....	24846-00
Clippers, Shears 7 <sup>1</sup> / <sub>4</sub> " .....	each .....	23694-00
Cylinder, graduated, poly, 25 mL .....	each .....	1081-40
Cylinder, graduated, poly, 100 mL .....	each .....	1081-42
Digesdahl Digestion Apparatus, 115 V .....	each .....	23130-20
Digesdahl Digestion Apparatus, 230 V .....	each .....	23130-21
Filter Discs, glass, 47 mm .....	100/pkg .....	2530-00
Filter Holder, membrane .....	each .....	2340-00
Filter Pump .....	each .....	2131-00
Flask, Erlenmeyer, 250 mL .....	each .....	505-46
Flask, filtering, 500 mL .....	each .....	546-49
Flask, volumetric, Class A, 50 mL .....	each .....	14574-41
Flask, volumetric, Class A, 100 mL .....	each .....	14574-42
Hot Plate, 4" diameter, 120 VAC .....	each .....	12067-01
Hot Plate, 4" diameter, 240 VAC .....	each .....	12067-02
pH Meter, <i>sens<sup>ion</sup></i> <sup>TM</sup> I, portable, with electrode .....	each .....	51700-10
pH Indicator Paper, 1 to 11 pH .....	each .....	391-33
Pipet Filler, safety bulb .....	each .....	14651-00
Pipet, serological, 2 mL .....	each .....	532-36
Pipet, serological, 5 mL .....	each .....	532-37
Pipet, TenSette, 0.1 to 1.0 mL .....	each .....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg .....	21856-96
Pipet Tips, for 19700-01 TenSette Pipet .....	1000/pkg .....	21856-28
Pipet, volumetric, Class A, 1.00 mL .....	each .....	14515-35
Spoon, measuring, 0.1 g .....	each .....	511-00

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



**Attachment 2**  
**Dilution of Water Samples for Hach Field Testing**  
**Standard Operating Procedure**

## **SOP – Dilution of Water Samples for Hach Field Testing**

### **Scope**

This operating procedure applies to Environmental Department/Federal Services Division projects. It is the procedure to be used for diluting samples of surface water or groundwater in the field so they can be analyzed using a Hach colorimeter. The procedure below applies only to inorganic constituents; some exceptions are noted below. Field personnel must be trained in this dilution procedure before attempting it in the field.

This procedure follows standard laboratory protocols. It requires laboratory-grade glassware, which is outlined below. It also requires careful use, care, and maintenance of this glassware to ensure accurate results.

### **Background**

Samples of surface and groundwater may be analyzed in the field to provide results that can help to guide sampling or to measure constituents with short holding times. Examples are measurement of hexavalent chromium or ferrous iron in groundwater (both of which have very short holding times) or sulfate and aluminum in surface or groundwater (which may be found in concentrations exceeding the upper range of the Hach instrument). In cases where sample concentrations exceed the upper range of the instrument, samples must be diluted so that a result can be measured. A dilution factor is then used to adjust the measured concentration to the actual concentration in the sample.

When possible, the results of previous sampling should be used to anticipate sample concentrations; these provide a starting point from which to estimate an appropriate dilution factor. In cases where there is little previous information, selecting an appropriate dilution factor can be hit or miss. Dilutions should be completed so that measured concentrations fall within the middle of the Hach test range where test accuracy is the highest.

### **Terminology**

**Actual or Real Concentration** – the concentration in an undiluted sample.

**Measured Concentration** – the concentration reported by a Hach instrument (equal to the actual concentration if the sample is not diluted).

**Dilution Factor** – a factor expressing the amount by which a sample is diluted prior to analysis (e.g., 10x is a dilution factor of 10).

**Meniscus** – the curvature of the upper surface of a fluid when contained within a cylinder due to surface tension/capillary forces along the cylinder walls; curvature increases with decreasing cylinder diameter.



mL – milliliter.

#### Minimum Required Equipment

Glassware	Other
Mohr glass pipet, 25 x 0.1 mL (2)	3 valve pipet filler
Mohr glass pipet, 10 x 0.1 mL (2)	Plastic cups (several)
250 mL glass beaker (2)	1 gallon commercial distilled water
100 mL glass beaker (6)	Nitrile gloves
50 mL glass beaker (4)	Field notebook
Stirring rods, glass	Hach reagents
	Hach colorimeter
	Paper towels

#### Equipment Use

**Important:** Before the first use of glassware, mark one 25 mL pipet, one 10 mL pipet, and one 250 mL beaker as “DI Only.” This glassware will only be used to deliver diluting distilled water; it should **NEVER** be used for samples. Mark the remaining pipets and 250 mL beaker as “Sample.”

The pipet tips are **extremely fragile** and should be carefully protected. The tip should always be placed inside the cardboard sleeve to prevent chipping when not in use. If the tip chips or breaks, the accuracy of the pipet is compromised and it must be replaced. Note frequent use of the term “gently” throughout this procedure; please adhere to this.

The Mohr pipets are calibrated to deliver fluid to the nearest 0.1 mL. Accurate reading of the meniscus is critical to producing consistent and accurate sample dilutions.

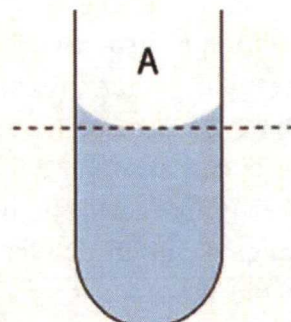
A volume of 0.1 mL is equivalent to one to two drops depending on the pipet; a practiced, gentle touch is required to dispense this small volume.



**Reading the Meniscus.** A meniscus is the curvature of a fluid when contained within a cylinder due to surface tension/capillary forces along the cylinder walls (Figure A). Figure A shows that the meniscus is properly read when the bottom of the meniscus rests on the required volume line and the volume line is held at eyelevel. Most pipets have major graduations shown as rings that encircle the pipet tube (for the 10 mL pipet this would be at 1 mL graduations). The pipet is at eyelevel when the ring appears as a single solid line (held too high or too low, the ring will appear as an oval).

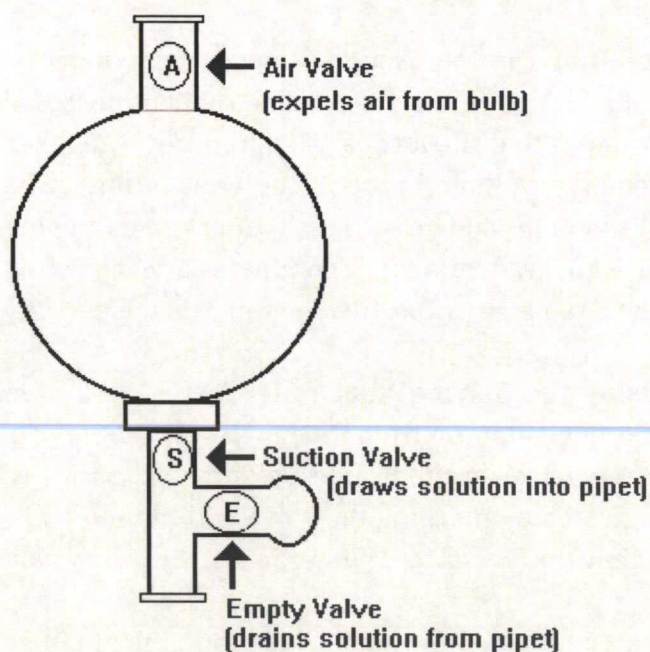
**Using the 3-Way Pipet Filler Valve.** A rubber pipet filler valve is used to draw and dispense solution from the pipets. A schematic of the valve is shown. The positions of the three valves are indicated by flattened circles with letters on the bulb stems. These are activated by pinching the valve with thumb and forefinger. The pipet attaches to the bulb below the "E" valve. With practice, the 3-way valve can be used with one hand.

**Valve "A"** is on the stem at the top of the bulb. Pressing valve "A" and squeezing the bulb expels air from the bulb. Pressing the valve when the bulb is collapsed allows the bulb to refill with air without affecting fluid levels in the pipet.





With the bulb collapsed, pressing and holding **Valve "S"** at the base of the bulb will suck solution into the pipet when the pipet tip is held below the surface of the solution (the bulb will slowly reinflate with air from the pipet). The rate at which solution is drawn into the pipet can be controlled by the amount of pressure applied to the valve. Care must be taken to keep the pipet tip below the solution surface as it draws down to prevent bubbling and the introduction of air into the pipet. Solution should be drawn into the pipet to a level slightly above the target volume (e.g., if you want to end with 5 mL, drawn in 6 mL; the excess will be discharged in the next step). Once the required volume has been drawn into the pipet, press valve "A" to fully inflate the bulb.



**Extreme care** must be taken to slowly draw solution so that it does not bubble into or otherwise get into the filler valve and bulb. If this occurs, the bulb must be decontaminated prior to its next use by flushing numerous times with DI water, then repeatedly filling the bulb and expelling air through each of the three valves until water droplets are no longer present.

Solution is dispensed from the pipet by pressing **Valve "E"** and applying gentle pressure to the bulb. Prior to dispensing the solution, the correct volume must be held within the pipet. To accomplish this, hold the pipet over the waste solution container at eyelevel as described above under Reading the Meniscus. Press valve "E" to allow solution to drip out of the pipet until the bottom of the meniscus rests on the desired volume. Any droplets hanging from the tip of the pipet should be removed by gently touching the tip to the side of the waste container (to prevent cross-contamination, do not touch the waste fluid!). Dabbing the tip on a paper towel should be avoided since this may suck solution out of the pipet tip which creates inaccuracy. Once the meniscus rests on the proper line, visually check the fluid level in the pipet tip. The solution should completely fill the pipet tip with no airspace.



Remember, the pipets are “calibrated to deliver.” This means that the pipet tip should be full and the meniscus should rest on the proper line. When dispensed, the pipet should be completely empty.

### **Dilution Procedure**

Prior to beginning sample dilutions, set one plastic cup to the side and label it as “Waste.” Fill two cups with distilled or deionized water. Label one cup “Rinse 1” and the other “Rinse 2.” Fill the glass beaker labeled “DI Only” with distilled or deionized water. Set several paper towels down on a flat surface to provide a clean working surface.

The best dilutions are made when extreme care is taken when implementing this procedure. Speed is not of the essence because it causes mistakes and inaccuracies. Trying your best to be accurate in all steps and with all procedures will ensure quality dilutions that are repeatable.

**Step 1.** Use knowledge of the Hach analytical range for the constituent you are measuring and previous analytical results, historic dilution records, or your best guess to determine the dilution factor you need to bring the sample concentration into the range of the Hach instrument. For ferrous or total iron the test range is 0.0 to 3.0 mg/L (although the instrument will read up to 3.3 mg/L); ideally you would want measured values of 0.5 to 2.5 mg/L. Also determine the volume of solution that you need (for iron, you’ll need 10 mL each for ferrous and total plus some volume for a margin of error, so 50 mL total solution is a good amount if you need 20 for the test; this way you’ll have enough to redo a test if an AccuVac ampule fails to fill sufficiently).

The next section shows how to calculate dilution factors.

**Step 2.** Using the sample pipet, carefully measure out the amount of sample you need. For example, if you are making 50 mL of diluted solution at a dilution factor of 10x, you need 5.0 mL of sample. Remove any hanging droplets and check to make sure that the tip is full. Discharge the sample volume into a clean 100 mL sample beaker.

For high dilution factors, where only a small amount of sample (<1 mL) is required, you may find it easier to fill the pipet part way (for example to 5.0 mL) then dispense the required volume. Experience indicates that higher head on the column of water in the pipet creates more consistent droplets as fluid is dispensed.

After delivery, immediately rinse the sample pipet by inserting into the Rinse 1 cup, and filling the pipet with rinse water to a level similar to or greater than the sample volume of sample dispensed. Discharge into the waste container and repeat. Then fully fill the pipet from the Rinse 2 cup and discharge into waste container. Finally, use the pipet bulb to blow air through the pipet to clear any residual rinse water that may remain. Remove the filler valve and carefully set the pipet aside where it will not roll away and so that the tip is protected (inserting the tip into the cardboard sleeve is a good idea).

**Step 3.** Using the pipet labeled DI Only, carefully measure out the amount of distilled or deionized water you need. Discharge into the 100 mL sample beaker taking care not to splash any of the solution. For the example above, you would need to add 45 mL of DI water (fill the 25 mL pipet to 25.0 mL, discharge and refill with 20.0 mL and discharge).

Use the pipet bulb to blow air through the pipet to clear any residual rinse water that may remain. Remove the filler valve and carefully set the pipet aside where it will not roll away and so that the tip is protected (inserting the tip into the cardboard sleeve is a good idea).

**Step 4.** Stir thoroughly using a clean glass rod. 100 times is a good number, 50 clockwise and 50 anticlockwise.

Rinse the stir rod by stirring a few times in first the Rinse 1 and then the Rinse 2 cup. Dry the rod on a clean paper towel and set aside where it will not roll away.

**Step 5.** Analyze the sample following the directions for the Hach protocol of interest. If using AccuVac ampules, these may be snapped off in the 100 mL sample beaker with no need to transfer the solution. Diluted solutions should be analyzed immediately after the dilution is completed.

**Step 6.** After completing the analysis, empty and decontaminate the 100 mL beaker using DI water. Dry thoroughly with a paper towel and set upside down on a clean paper towel.

### Calculations

The dilution factor equals the final sample volume divided by the initial sample volume. For the 10x dilution example above,  $DF = 50 \text{ mL} / 5 \text{ mL} = 10$ .

Serial dilutions (a two-step process in which you further dilute a diluted solution) can be used to achieve dilution factors of 10,000 or higher if the need arises.

The following table shows the required volumes of sample and DI water to produce 50 mL of diluted solution under different dilution factors.

<b>Dilution Guide to Produce 50 mL of Final Volume</b>		
<b>Dilution Factor</b>	<b>Sample Volume (mL)</b>	<b>DI Volume (mL)</b>
1	50	0
2	25	25
5	10	40
10	5	45
20	2.5	47.5
25	2	48
50	1	49
100	0.5	49.5
500	0.1	49.9
<b>Step 2 serial dilutions using 500x sample dilution as the sample</b>		
1,000	25 mL 500x dilution	25
5,000	5 mL 500x dilution	45
10,000	2.5 mL 500x dilution	47.5

To calculate the actual analyte concentration from a diluted sample:

$$\text{Actual Concentration (mg/L)} = \text{Measured Concentration (mg/L)} \times \text{Dilution Factor}$$

Example:

A previous analysis of well water measured a total iron concentration of 1,250 mg/L. What is an appropriate dilution factor?

$1,250 \text{ mg/L} / 3 \text{ mg/L} = 416.7$ , where 3 mg/L is the upper range of the Hach test. A dilution factor of 500 should produce a measured result of 2.5 and would be an appropriate factor to use.

**Required Notes and Observations**



The following should be recorded in the field notebook:

- Sample id number
- Date and time of collection
- Date and time of dilution and analysis
- Volume of sample used
- Volume of distilled or deionized water used
- Dilution factor
- Hach analytical result
- Actual sample concentration
- Any problems or uncertainties regarding dilution volumes and measurements
- Any interferences or problems with the Hach test.

#### **Other Important Considerations**

The technique described above has produced field measurements of total iron with a correlation coefficient exceeding 0.95 compared to laboratory analytical results across 6 orders of magnitudes of iron concentration. It is the same technique used by laboratories adapted to field use.

- Provide yourself with a clean, open working environment so that you can organize your glassware and keep things clean. The back of an SUV works fine, a motel room is better.
- Practice makes perfect. With experience, sample dilutions can be accomplished confidently in a few minutes' time. Running batches of a few samples at a time will increase efficiency.
- **Before mobilizing to the field**, check to make sure all glassware is clean and that pipet tips and beakers are not chipped, cracked, or otherwise compromised. Replace any that are.
- **Before mobilizing to the field**, test a sample of your distilled or deionized water as a blank sample to ensure that it does not give a positive result on the Hach test. If it does, you need to obtain different water for diluting your samples.
- Cleanliness is paramount. Be sure to clean sample pipets and beakers thoroughly and be vigilant in maintaining separation between glassware used for DI water and

samples to prevent cross-contamination. **Never, ever** put dirty glassware away, store it clean and dry.

- Know the Hach procedure you are using, including potential interferences that may occur as a result of dilution.
- Take the time to run duplicates to check your consistency in performing dilutions, record duplicates in the log book.
- Take the time to look at laboratory analytical results to check your accuracy.
- Be safe rather than sorry. If there are ever doubts or questions regarding the meniscus reading, volume delivered by the pipet, or any other aspect of the dilution (e.g., formation of a precipitate), dump what you are doing, clean the glassware, and start again.
- For most metals, both deionized and commercial distilled waters should produce acceptable results. This may not be true for anions (e.g., sulfate, carbonate) and some volatile metals which may be present in distilled water. Many bottled drinking waters such as Dasani or Aquafina have cations and anions added for flavor; these and other drinking waters should **not** be used for dilutions.
- Dilution may potentially cause minor changes to redox-sensitive species such as ferrous iron or hexavalent chromium. Because water has only a few mg/L of dissolved oxygen, changes in oxidation state resulting from the addition of DI water are likely to be minimal. However, the process of stirring increases contact with the atmosphere which has an oxygen concentration of about 20 percent. When stirring dilutions for redox-sensitive species, care should be taken to minimize splashing and unnecessary contact with air.
- To prevent the oxidation of redox-sensitive species, collect samples with no free headspace and analyze as quickly as possible.
- For analyses of ferrous and total iron, the geochemical parameter of interest is typically the ferrous/total iron ratio, rather than the absolute concentration of ferrous iron. To avoid errors that might be introduced by dilution, both the ferrous and total iron ampules should be filled from the same diluted sample (50 mL is sufficient to accomplish this). In this manner, the ratio will be correct even if minor errors are introduced to the absolute concentrations.